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TITLE: NONAQUEOUS ELECTROLYTE LITHIUM SECONDARY BATTERY

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COUNTRY

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N/A

APPL-NO: JP06122401

APPL-DATE: June 3, 1994

INT-CL (IPC): H01M010/40

ABSTRACT:

PURPOSE: To provide a lithium secondary battery excellent in capacity for large current discharging and low temperature discharging, without worsening charging/discharging efficiency at a room temperature, discharging capacity and volumetric energy density, in the lithium secondary battery using a material of graphite system in a negative electrode carbon material.

CONSTITUTION: A nonaqueous electrolyte lithium secondary battery, comprising a positive electrode with a lithium compound oxide containing at least one kind of transition metal serving as an active material, negative electrode with a carbon material mainly contained with graphite serving as an active material, electrolyte fusing at least  $\text{LiPF}_6$  in an organic solvent as the electrolyte and a separator, is provided. This organic solvent contains ethylene carbonate, dimethyl carbonate and diethyl carbonate, and in this composition ratio, when represented x, y, z (unit volume %) respectively for ratio of ethylene carbonate, dimethyl carbonate and diethyl carbonate relating to a total sum of ethylene carbonate, dimethyl carbonate and diethyl carbonate, a relation is  $0 < x \leq 25$ ,  $45 \leq y < 100$ , further  $0 < z < y$ .

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DERWENT-ACC-NO: 1996-083959

DERWENT-WEEK: 199609

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TITLE: Lithium secondary battery with non-aqueous electrolyte -  
using organic solvent contg. ethylene carbonate,  
di:methyl carbonate and di:ethyl carbonate

PATENT-ASSIGNEE: SUMITOMO CHEM CO LTD[SUMO]

PRIORITY-DATA: 1994JP-0122401 (June 3, 1994)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 07335254 A	December 22, 1995	N/A	006	H01M 010/40

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
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INT-CL (IPC): H01M010/40

ABSTRACTED-PUB-NO: JP 07335254A

BASIC-ABSTRACT:

The battery has a positive electrode is made of an active material like an oxide of lithium compound so that at least one transition metal exists. The negative electrode contains an active carbon material which mainly contains graphite. An organic solvent having LiPF<sub>6</sub> solution is used as the electrolyte. A separator is provided in the battery.

The organic solvent contains ethylene carbonate, dimethyl carbonate and diethyl carbonate whose unit volume percentage are expressed as  $0 < x \leq 25$ ,  $45 \leq y < 100$  and  $0 < z < y$  where x=ethylene carbonate, y=dimethyl carbonate and z=diethyl carbonate.

ADVANTAGE - Improves efficiency of battery. Increases capacitive characteristics.

CHOSEN-DRAWING: Dwg.0/1

TITLE-TERMS: LITHIUM SECONDARY BATTERY NON AQUEOUS ELECTROLYTIC ORGANIC SOLVENT  
CONTAIN ETHYLENE CARBONATE DI METHYL CARBONATE DI ETHYL CARBONATE

DERWENT-CLASS: L03 X16

CPI-CODES: L03-E01C; L03-H05;

EPI-CODES: X16-B01F1; X16-J02; X16-J08;

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C1996-027114

Non-CPI Secondary Accession Numbers: N1996-070329

EC 25

DMC 45-99  
DEC

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the lithium secondary battery which uses nonaqueous electrolyte.

[0002]

[Description of the Prior Art] Alpha-NaFeO<sub>2</sub> The positive electrode which uses as an active material the transition-metals oxide which uses as a parent the stratified lithium multiple oxide or spinel type structure which uses mold structure as a parent. The lithium secondary battery using the negative electrode which uses as an active material a dope and the carbon material which can be dedoped a lithium. Since per volume and the energy density per weight are high, and a miniaturization and lightweight-izing are easy and it excels in charge-and-discharge cycle nature and safety, The big spotlight as rechargeable batteries for portable electrical machinery and apparatus, such as a cellular phone and a pocket type video camera, or a rechargeable battery of an electric vehicle application is captured.

[0003] However, by one side, the badness of a high current discharge property is mentioned as a big trouble of a lithium secondary battery. This has the high activity of the cell active material used in the lithium secondary battery, and is because the solvent for the electrolytic solutions is asked for high oxidation resistance and reducibility-proof, so a non-aqueous solvent must generally be used. since [ namely, ] nonaqueous electrolyte is used for the electrolytic solutions, such as a conventional manganese cell, a conventional nickel cadmium cell, etc., with the lithium secondary battery as compared with the drainage system cell which uses the water solution -- the electric conductivity of the electrolytic solution -- at most -- it is 10 mS/cm extent and this is a low value of 1/10 or 1/100 as compared with the drainage system electrolytic solution. therefore, the ohmic loss of the lithium secondary battery using nonaqueous electrolyte was markedly boiled as compared with the cell which uses the drainage system electrolytic solution, and had the trouble of being large.

[0004] It is one side, and in order to improve the capacitance characteristics of a cell further, examination of the cell active material itself is also performed, especially in the case of the carbon material of a negative electrode, the charge-and-discharge capacity per unit weight is large, and it is pointed out that the average potential in charge and discharge is the ingredient the graphite system ingredient excelled [ ingredient ] in the point that it is low and energy density is large. However, when a graphite system ingredient is used as a carbon material of a negative electrode, in order for a negative electrode to decompose the propylene carbonate conventionally used as nonaqueous electrolyte for lithium cells at the time of cell charge, generally ethylene carbonate is used. However, about the low-temperature discharge property of the lithium secondary battery using the electrolytic solution containing ethylene carbonate, not a not necessarily satisfying property but still much more improvement in a property is called for.

[0005] From such a situation, the electrolytic solution made to dissolve lithium salt in the mixed solvent of annular carbonate and un-annular carbonate as the electrolytic solution of a lithium cell was proposed

according to that reactivity with an electrode is low, and the reason electric conductivity is comparatively high (JP,4-171674,A).

[0006] When a graphite system carbon material is used for a negative-electrode ingredient among a carbonate system solvent especially, it excels in all of the high current discharge property after the first stage and preservation. And noting that it is the nonaqueous electrolyte excellent also in the cycle property and excels further in both the bottom high current discharge property of ordinary temperature, and a low-temperature discharge property The mixed solvent of three components of ethylene carbonate, dimethyl carbonate, and diethyl carbonate was contained, and the electrolytic solution of the range of 30 volume % - 70 volume % was proposed for the content of ethylene carbonate (JP,5-013088,A).

[0007] Furthermore, the electrolytic solution whose presentation ratio of diethyl carbonate the electrolytic solution with the presentation whose ion conductivity of the electrolytic solution improved for the purpose of improvement in a low-temperature property is examined, and the presentation ratio of ethylene carbonate and dimethyl carbonate is 1:1 in a volume ratio, and is ten to 33 volume [ of the whole solvent ] % was proposed (the content of JP,5-283104,A and the ethylene carbonate in this case is equivalent to 33 - 45 volume %).

[0008]

[Problem(s) to be Solved by the Invention] However, as a result of examining the electrolytic solution of the presentation by which the conventional proposal was made, only a discharge capacity lower than the discharge capacity expected from the electric conductivity of the -20-degree C electrolytic solution was obtained, but it turned out that a low-temperature discharge property is not still more enough, either. The purpose of this invention is in the lithium secondary battery which used the ingredient of a graphite system for the negative-electrode carbon material to offer the lithium secondary battery excellent in the capacitance characteristics in high current discharge and low-temperature discharge, without spoiling the effectiveness of the charge and discharge in a room temperature, discharge capacitance characteristics, and volume energy density.

[0009]

[Means for Solving the Problem] As a result of seeing such a situation and this invention persons' inquiring wholeheartedly, when there are three components of the ethylene carbonate which is the organic solvent of the electrolytic solution, dimethyl carbonate, and diethyl carbonate in specific presentation within the limits, it came to complete a header and this invention for said technical problem being solvable.

[0010] That is, this invention is invention described below.

(1) The positive electrode which used the lithium multiple oxide containing at least one sort of transition metals as the active material, It is [ the negative electrode which used as the active material the carbon material which mainly contains a graphite, and ] LiPF<sub>6</sub> at least as an electrolyte. In the nonaqueous electrolyte lithium secondary battery equipped with the electrolytic solution which dissolved in the organic solvent, and a separator This organic solvent contains ethylene carbonate, dimethyl carbonate, and diethyl carbonate. Ethylene carbonate [ as opposed to total of ethylene carbonate, dimethyl carbonate, and diethyl carbonate in the presentation ratio ], The nonaqueous electrolyte lithium secondary battery characterized by being  $0 < x \leq 25$ ,  $45 \leq y < 100$ , and  $0 < z < y$  when the rate of dimethyl carbonate and diethyl carbonate is expressed as x, and y and z (unit volume %), respectively.

[0011] (2) LiPF<sub>6</sub> in the electrolytic solution Nonaqueous electrolyte lithium secondary battery given in (1) characterized by a density range being 0.5-1.5 mols/l.

[0012] Next, this invention is explained to a detail. The nonaqueous electrolyte lithium secondary battery of this invention is [ the positive electrode which used the lithium multiple oxide containing at least one sort of transition metals as the active material, the negative electrode which used as the active material the carbon material which mainly contains a graphite, and ] LiPF<sub>6</sub> at least as an electrolyte. It has the electrolytic solution which dissolved in the organic solvent, and a separator. In the nonaqueous electrolyte lithium secondary battery of this invention, the solvent used for the electrolytic solution is characterized by being  $0 < x \leq 25$ ,  $45 \leq y < 100$ , and  $0 < z < y$ , when the presentation ratio expresses the rate of the ethylene carbonate to total of ethylene carbonate, dimethyl carbonate, and diethyl carbonate,

dimethyl carbonate, and diethyl carbonate as x, and y and z (unit volume %) including ethylene carbonate, dimethyl carbonate, and diethyl carbonate, respectively. These rates are  $0 < x \leq 20$ ,  $50 \leq y < 100$ , and  $0 < 2z < y$  preferably. If the electrolytic solution in which the presentation rate of ethylene carbonate has the presentation exceeding 25 volume % is used, since the discharge capacity in low temperature will not become large, it is not desirable.

[0013] Although it is not yet clear about this cause, when the viscosity of the electrolytic solution increases at the time of low temperature and a direct current is passed like a cell so that the content of ethylene carbonate with a high freezing point is large, it is easy to produce the concentration ununiformity of the ion near the electrode at the time of discharge, and thinks because the effect of concentration polarization became large relatively. Since the discharge capacity in low temperature and the discharge capacity at the time of high current discharge fall, the presentation rate of the dimethyl carbonate which is un-annular carbonate is not desirable at under 45 volume %. About the presentation rate of the diethyl carbonate which is un-annular carbonate, since the discharge capacity at the time of high current discharge falls especially above the rate of diethyl carbonate, and an EQC, it is not desirable.

[0014] The lithium multiple oxide which contains at least one sort of transition metals as an active material is used for the positive electrode in the nonaqueous electrolyte lithium secondary battery of this invention. As this positive electrode, after mixing the binder for being settled in the powder of this lithium multiple oxide, auxiliary electric conduction agent powder, and these powder etc. to homogeneity, pressing is carried out, or it pastes using a solvent etc., it presses after spreading desiccation on a charge collector, and, specifically, the thing of a configuration of having fixed on the charge collector sheet is mentioned.

[0015] The lithium multiple oxide in this positive electrode which contains at least one sort of transition metals, such as vanadium, manganese, iron, cobalt, and nickel, as a lithium multiple oxide containing at least one sort of transition metals is mentioned. It is  $\alpha$ - $\text{NaFeO}_2$  of transition metals, such as cobalt and nickel, at the point desirable about especially that average discharge potential is high. The lithium multiple oxide which uses as a parent spinel type structure of transition metals, such as a stratified lithium multiple oxide which uses mold structure as a parent, or manganese, is mentioned. In that the cycle property is preferably excellent especially, the stratified lithium multiple oxide which makes a lithium nickel multiple oxide a subject is mentioned.

[0016] As auxiliary electric conduction material powder used for this positive electrode, there is the electric conduction effectiveness and a conductive polymer etc. is mentioned in graphite powder, carbon black, and the end of a coke breeze that what is necessary is just what has the resistance over the electrochemical reaction in the resistance, positive electrode, and negative electrode to the nonaqueous electrolyte to be used. As for the amount of this auxiliary electric conduction material, it is desirable to consider as 1 - 20 weight section extent to the active material powder 100 weight section to be used.

[0017] The carbon material which mainly contains a graphite as an active material is used for the negative electrode in the nonaqueous electrolyte lithium secondary battery of this invention. The rate of the graphite in this carbon material has 70 desirable % of the weight or more, and its 90 % of the weight or more is still more desirable. Carbon black, corks, etc. are mentioned as carbon materials other than a graphite. After mixing to the binder etc. and homogeneity for binding these powder to the carbon material powder which specifically contains a graphite mainly further as a negative electrode in this invention, pressurization molding is carried out, or it pastes using a solvent etc., it presses after spreading desiccation on a charge collector, and the thing of a configuration of having fixed on the charge collector sheet is mentioned.

[0018] A natural graphite and an artificial graphite are mentioned as this graphite. As this graphite, the lattice spacing (d002) in an X diffraction is 3.37Å or less, and, specifically, 2.23 or more things have desirable true specific gravity. The lattice spacing (d002) in an X diffraction is 3.36Å or less still more preferably, and true specific gravity is 2.24 or more things. A lattice spacing (d002) means the thing of the value measured by the X-ray diffraction method [the Otani \*\*\*\*, a carbon fiber, and a 733 - 742-page (1986) modern edit company] which uses the high purity silicon as the standard substance here,

using CuK alpha rays as an X-ray.

[0019] The ash content of the graphite used in this invention is 0.1 or less % of the weight more preferably 0.5 or less % of the weight. In the case of a natural graphite, it changes also with places of production, but since the ash content to contain is as large as several % of the weight or more, 2500 degrees C or more of things which processed by high temperature 2800 degrees C or more still more preferably, and carried out ash content to 0.1 or less % of the weight more preferably 0.5 or less % of the weight are preferably good. Ash content means the value by JISM8812 here.

[0020] As an artificial graphite used in this invention, a scale-like graphite (the product made from SEC, trade names SGP5, SGP15, and SGO5, the product made from SGX5; LONZA, trade names SFG6, SFG15, KS6, and KS15), nodular graphite (the Osaka Gas Co., Ltd. make, the trade names [ MCMB / MCMB and / 20-28 ] 6-28), a fibrous graphite (the Osaka Gas Co., Ltd. make, trade names SG241 and F500), etc. can be illustrated, for example. Although especially the grain size of the carbon material which mainly contains the graphite used in this invention is not restricted, that whose mean particle diameter is about 1-50 micrometers is desirable. It is 2-20 micrometers still more preferably.

[0021] As a binder used for an aforementioned positive electrode and an aforementioned negative electrode, there is the binding effectiveness and a fluororesin, polyethylene, a polypropylene, etc. are mentioned that what is necessary is just what has the resistance over the electrochemical reaction in the resistance, positive electrode, and negative electrode to the nonaqueous electrolyte to be used. As for the amount of this binder, it is desirable to consider as 1 - 20 weight section extent to the active material powder 100 weight section to be used.

[0022] Nickel, titanium, stainless steel, copper, aluminum, etc. are mentioned that what is necessary is just what has the resistance over the electrochemical reaction in the resistance, positive electrode, and negative electrode to the nonaqueous electrolyte to be used as a charge collector used for an aforementioned positive electrode and an aforementioned negative electrode. The thickness of this charge collector is so desirable that it is thin as long as reinforcement maintains in that the volume energy density as a cell goes up, and its about 5-100 micrometers are desirable. As a charge collector of this positive electrode, it is easy to process it into a thin film, and aluminium foil is desirable at the point of being cheap. Copper foil is desirable at the point referred to as being hard to make a lithium and an alloy and being easy to process it into a thin film as a charge collector of this negative electrode.

[0023] In the nonaqueous electrolyte lithium secondary battery of this invention, as a separator, contact of two poles is prevented, and it has insulation, and nonaqueous electrolyte is held, it has the function which can penetrate a lithium ion and nonwoven fabrics, such as olefin system resin, such as fluororesin, polyethylene, and polypropylene, and nylon, and textile fabrics can be illustrated that what is necessary is just what has the resistance over the electrochemical reaction in the resistance, positive electrode, and negative electrode to the nonaqueous electrolyte to be used. The thickness of this separator is so good that it is thin as long as mechanical reinforcement is maintained at the point said that the volume energy density as a cell goes up, and internal resistance becomes small, and its about 10-200 micrometers are desirable.

[0024]

[Example] Hereafter, although an example explains this invention to a detail further, this invention is not limited at all by these.

(I) Nickel acid lithium powder which mixed nickel carbonate with the lithium nitrate, calcinated for 15 hours and was obtained at 700 degrees C in the oxygen air current as a specification positive electrode of the lithium secondary battery with which the trial was presented, Artificial-graphite powder and the mixed powder which set polyvinylidene fluoride to 87:10:3 by the weight ratio N-methyl pyrrolidone solution was distributed, it considered as the slurry, the after [ spreading ] vacuum drying was carried out on aluminum foil, and what cut down the sheet-like electrode pressed and produced in 1.5cmx2.0cm magnitude was used. When X diffraction measurement was performed for the obtained nickel acid lithium powder, it is alpha-NaFeO<sub>2</sub>. Having mold structure was checked.

[0025] The scale-like natural-graphite powder from Madagascar processed by 3000-degree-C processing as a negative electrode, Make N-methyl pyrrolidone solution distribute carbon black (Tokai Carbon Co.,

Ltd. make) and the mixed powder which set polyvinylidene fluoride (the Kureha chemistry company make) to 86:4:10 by the weight ratio, and it considers as a slurry. It dried after applying on copper foil, and what cut down the sheet-like electrode pressed and produced in 1.5cmx2cm magnitude was used. As a separator, the polypropylene porosity film (die cel chemistry company make, trade name Celgard #2240) was used.

[0026] (II) -- charge and discharge test (1) charge: -- charge always carried out charge maximum electrical-potential-difference  $V_{\max} = 4.1V$ , 5mA, and constant current constant-potential charge of 3 hours at 20 degrees C.

(2) Room temperature spark test : in 20 degrees C, the spark test was carried out by constant current (cut-off voltage 2.75V and 1.5mA). This hits the discharge conditions of 0.2C.

(3) Low-temperature spark test : in -20 degrees C, the spark test was carried out by constant current (cut-off voltage 2.75V and 1.5mA).

(4) High current spark test : in 20 degrees C, the spark test was carried out by constant current (cut-off voltage 2.75V and 15mA). This hits the discharge conditions of 2C.

[0027] The mixed solvent which consists of the ethylene carbonate (EC) of the presentation shown in Table 1, dimethyl carbonate (DMC), and diethyl carbonate (DEC) as the example 1 electrolytic solution is used, and it is LiPF<sub>6</sub> as an electrolyte. What was prepared so that it might become in l. and one mol /was used. The positive electrode and negative electrode which were obtained as mentioned above were made to counter through a separator, it contained in the container made from stainless steel, and the cell A1 was produced. The discharge capacity of the obtained cell was about 7 mAh(s).

[0028] The electrolytic-solution presentation ratio and discharge capacity of the produced cell are shown in Table 1. Room temperature discharge capacity is the discharge capacity which converted the discharge capacity in the room temperature spark test of the above (2) at the room temperature of the produced cell into per positive-active-material weight among Table 1. However, with low-temperature discharge capacity retention It is a rate to the discharge capacity in the room temperature spark test of the above (2) of the discharge capacity in the low-temperature spark test of the above (3), and high current discharge capacity retention is the rate of the discharge capacity in the room temperature spark test of the above (2) of the discharge capacity in the high current spark test of the above (4).

[0029]

[Table 1]

	電池	混合溶媒組成比 (体積%)	室溫放電容量 (mAh/g)	低溫放電容量 保持率 (%)	大電流放電容量保持率 (%)	
		EC:DMC:DEC	0.2C	0.2C	1C	2C
実施例 1	A1	20:60:20	146	83	89	52
比較例 1	R1	60:30:10	150	<10		
	R2	50:40:10	150	<10	79	41
	R3	40:40:20	144	11	68	30
	R4	40:50:10	137	10	65	29
	R5	20:40:40	140	83	73	35
	R6	30:30:40	143	59	68	31
	R7	30:50:20	142	63	79	43
	R8	30:60:10	141	67	77	44
	R7	20:20:60	137	21	54	20

The relation between the amount of discharge in 20 degrees C of the cell A1 concerning this invention

and -20 degrees C and cell voltage is shown in drawing 1 .

[0030] The mixed solvent which consists of the ethylene carbonate (EC) which has the presentation ratio shown in Table 1 as the example of comparison 1 electrolytic solution, dimethyl carbonate (DMC), and diethyl carbonate (DEC) is prepared, and the all are LiPF<sub>6</sub> as an electrolyte. What was prepared was used so that it might become in 1. and one mol / , and also cells R1-R9 were produced like the example 1. A charge and discharge test unites and shows the electrolytic-solution volume composition ratio and discharge capacity of the cell which performed like the example 1 and was produced in Table 1. The relation between the amount of discharge in 20 degrees C of the comparison cell R4 and -20 degrees C and cell voltage is shown according to drawing 1 .

[0031] the voltage drop according [ both this invention cell A1 and the comparison cell R4 ] to the resistance component of immediately after discharge and the electrolytic solution as shown in drawing 1 -- said -- \*\*\*\*\* -- having . This can be explained from the electric conductivity in -20 degrees C by 1kHz alternating current measurement of the electrolytic solution used for the cell A1 and the cell R4 being the same. However, during discharge, to a room temperature and the inclination of a -20-degree C discharge curve not changing mostly, the inclinations of a discharge curve differ and it turns out by the comparison cell R4 that the comparison cell R4 of descent of potential is earlier at a room temperature and -20 degrees C this invention cell A1. When the electrolytic solution of the presentation by which the conventional proposal was made is used, at the time of discharge at low temperature, it is shown the overvoltage is large rather than being expected from the electric conductivity of the electrolytic solution.

[0032]

[Effect of the Invention] The nonaqueous electrolyte lithium secondary battery of this invention is excellent in the capacitance characteristics in high current discharge and low-temperature discharge, without spoiling the effectiveness of the charge and discharge in a room temperature, discharge capacitance characteristics, and volume energy density. Since the lithium secondary battery of this invention maintains a big discharge capacity also in the -20-degree C cold district which is generally the service temperature minimum of a lithium secondary battery and the extreme fall of discharge capacity does not bring it about at the time of high current discharge of 1 more or more C, either, its industrial value is large as transportation device applications, such as pocket electronic equipment used out in the fields etc. or an electric vehicle.

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[Translation done.]



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CLAIMS

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[Claim(s)]

[Claim 1] The positive electrode which used the lithium multiple oxide containing at least one sort of transition metals as the active material, It is [ the negative electrode which used as the active material the carbon material which mainly contains a graphite, and ] LiPF<sub>6</sub> at least as an electrolyte. In the nonaqueous electrolyte lithium secondary battery equipped with the electrolytic solution which dissolved in the organic solvent, and a separator This organic solvent contains ethylene carbonate, dimethyl carbonate, and diethyl carbonate. Ethylene carbonate [ as opposed to total of ethylene carbonate, dimethyl carbonate, and diethyl carbonate in the presentation ratio ], The nonaqueous electrolyte lithium secondary battery characterized by being  $0 < x \leq 25$ ,  $45 \leq y < 100$ , and  $0 < z < y$  when the rate of dimethyl carbonate and diethyl carbonate is expressed as x, and y and z (unit volume %), respectively.

[Claim 2] LiPF<sub>6</sub> in the electrolytic solution Nonaqueous electrolyte lithium secondary battery according to claim 1 characterized by a density range being 0.5-1.5 mols/l.

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[Translation done.]